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(54) **Oil regenerating membrane**

(57) An oil regenerating membrane consists essentially of 5—95 parts by weight of fibrous paper pulp material and 95—5 parts by weight of adsorbent (e.g. granular or powdery activated clay, acid clay, diatomaceous earth, zeolite, silica gel, calcium oxide or activated carbon) per 100 part by weight of membrane. The membrane may be of cup-like form with two sheets 1 and 1′ adhesively bound together at their sides and bottom by a binding material 2 of regenerated cellulose. Hot oil, e.g. edible frying oil, is poured into the cup and permeates therethrough. Solids are filtered out and odoriferous impurities are adsorbed.

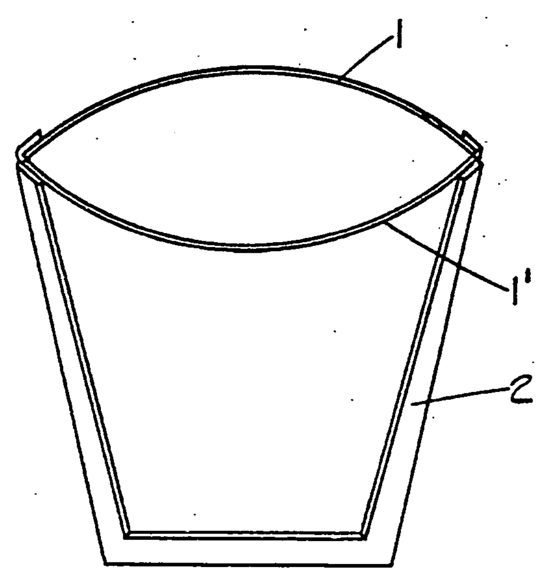

FIG.3.

The drawing originally filed was informal and the print here reproduced is taken from a later filed formal copy.

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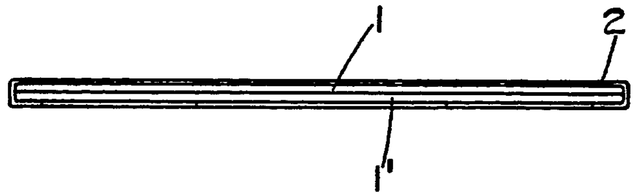

FIG. 1.

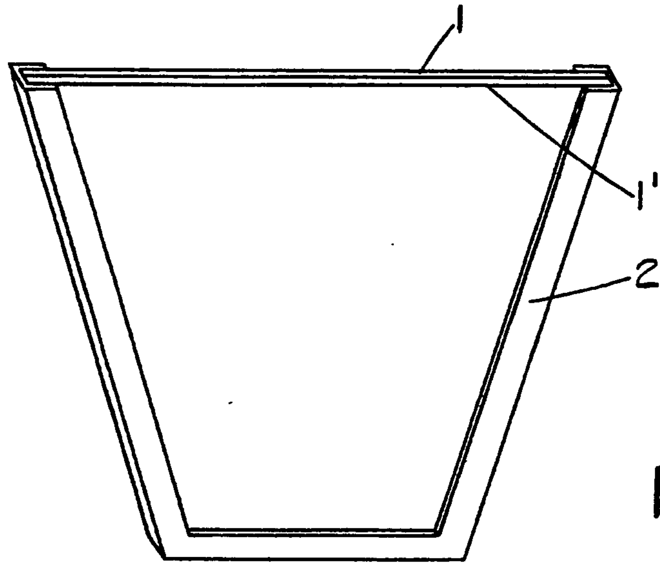

FIG. 2.

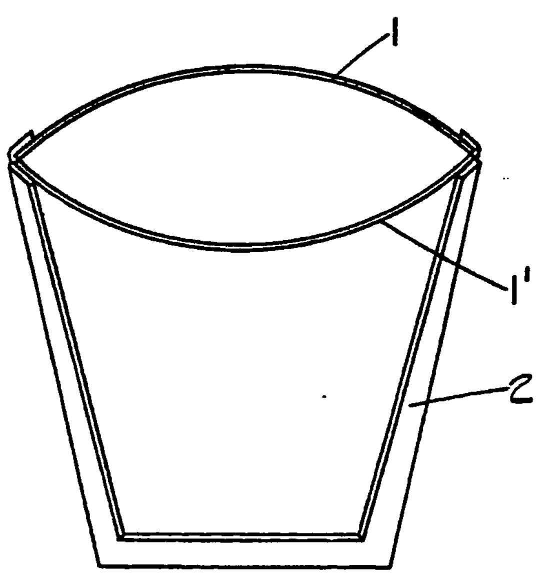

FIG. 3.

SPECIFICATION
Oil regenerating membrane

This invention relates to an oil regenerating filtration membrane and more particularly, but not exclusively to an edible oil regenerating membrane for household use or small scale business use for regenerating deteriorated oil by adsorbing and filtering off impurities produced as a result of deterioration of the used frying oil.

Edible oil used for frying fritters, chips etc., is deteriorated by chemical changes such as oxidation, polymerization, etc., and repeated use of a frying oil results in spoiling the taste of cooked products due to transfer of smells to the oil from fried materials such as fish or meat.

Conventional commercially available edible oil regenerating devices for household use for regenerating used frying oil containing deteriorating ingredients as described above have been primarily intended to filter off solids in the used edible oil, and therefore deterioration products dissolved in the oil and odors are scarcely removed.

As a result of extensive investigations, it has now been discovered that an adsorbent filtration membrane made by unifying fibrous paper pulp material and an absorbent can solve the above-described problems, eliminates the necessity of adding a powdery fine dust to the oil, and thus enables purification of the used oil without stirring.

Thus, the present invention is an oil regenerating filtration membrane consisting essentially of from 5 to 95 parts by weight of fibrous paper pulp material and from 95 to 5 parts by weight of adsorbent per 100 parts by weight of membrane. In the accompanying drawings:—

Figures 1 and 2 illustrate views of a trapezoidal filter cup according to the invention, and Figure 3 illustrates a perspective view of the same filter cup opened for use.

As is known, oils typically have such a high viscosity that their filtration rate through a membranous filter medium such as filter paper or filter cloth is extremely low at an ordinary temperature (e.g., about 40°C or less). At an elevated temperature (e.g., about 60°C or more), however, they usually undergo reduction in viscosity, resulting in accelerated filtration rate.

However, experiments by the inventors have revealed that the degree of purification of oil by adsorption increases as the temperature of the oil becomes higher. This temperature dependence of the adsorption rate may be attributable to the change in permeability of oil into an adsorbent, which depends upon the viscosity of the oil.

The inventors have discovered that this improvement in adsorption purification rate by an increase in the oil temperature overcomes the problems resulting from the increase in the filtration rate, and therefore that a heated oil immediately after used for frying can be sufficiently purified by passing it only once through an adsorbent layer made in a membrane form, thus having achieved the present invention.

Additionally, in the case of using an absorbent such as activated clay or diatomaceous earth as powder for the purification of edible oil, there must be, separately disposed, a tank for oil used, an adsorption tank, a filter, and a purified oil receiver, and hence the system is inevitably large-sized. Thus, such a system is difficult to make compact for the treatment of regenerating a comparatively small quantity of used oil as in a household or small business.

More particularly, when a deteriorated oil having been used for making fritters is placed, for example, in a cup-like form of a filtration membrane of the present invention, the oil permeates through the porous outer wall to undergo filtration, during which deterioration products in the oil and odorous compounds are adsorbed and solids such as fried chips and carbonized materials are filtered off, thus a purified oil being obtained as a filtrate.

As the adsorbent to be used in the present invention, porous inorganic solid adsorbents such as activated clay, acid clay, diatomaceous earth, aluminum hydroxide gel, activated alumina, silica gel, zeolite, calcium oxide, and activated carbon can be used. These can be used alone or in combinations of two or more. The particle size of the granular or powdery adsorbent is not particularly limited but, usually, those having an average particle size (average diameter) of 500 μm or less are preferred.

Of the above-described adsorbents, montmorillonite series adsorbents such as activated clay and acid clay are the most preferred because they possess a large oil regenerating ability and are obtained inexpensively. However, they have a peculiar odor, and, in some cases, the odor transfers to the oil. However, it has now been found that such odor is often based on acidic materials such as hydrogen sulfide, which can be removed by treating the clays with an aqueous solution of an alkaline material. Therefore, montmorillonite series adsorbents for use in the present invention are preferably treated with an aqueous alkaline material solution. As the alkaline material, for example, sodium hydroxide, potassium hydroxide, calcium hydroxide, sodium carbonate, potassium carbonate, potassium bicarbonate, and sodium bicarbonate, can be used. These may be used alone or in combination. The pH for the treatment is not particularly limited, but the pH of the filter cake obtained by neutralizing an aqueous suspension of a montmorillonite series material with an alkaline material aqueous solution and filtering the resulting suspension (and, if necessary, further neutralizing with an acid (for example, hydrochloric acid) followed by washing with water) is preferably 7.0 or less and, more preferably, from 3.0 to 6.0. In case of adding an alkali to a clay and further adding an acid thereto, it is not preferred that the pH at addition of alkali is above 9.0. If the pH at addition of alkali is 7 or less, it is not particularly

necessary to neutralize with an acid.

Fibrous paper pulp material to be used in the present invention comprises natural or synthetic fiber and is not particularly limited as long as it has sufficient heat resistance and safety in view of food sanitation considerations. However, vegetable-based fibers of high purity and toughness, such as cotton, hemp, rayon, etc., are preferable. Thin, long, and hydrophilic fibers are preferable. The fibrous paper pulp material to be used in the present invention is not necessarily of a single kind, and two or more fibrous paper pulp materials may be used in combination.

The oil regenerating filtration membrane to be used in the present invention has a composition of from 5 to 95 parts by weight fibrous paper pulp material and from 95 to 5 parts by weight of adsorbent. Membranes comprising from 20 to 40 parts by weight fibrous paper pulp material and from 80 to 60 parts by weight of adsorbent are generally preferred, as showing a good oil regenerating efficiency, although this depends upon the kinds of fibrous paper pulp material and adsorbent used.

The oil regenerating filtration membranes of the present invention are generally prepared by mixing fibrous paper pulp materials and adsorbents in water to form a slurry and filtering the slurry with a porous metallic mold or a mesh to mold a membrane, and are not limited to those having a uniform layer of fibrous paper pulp material and adsorbent, and include those of multi-layered structure having two or more layers different from each other in the composition ratio of fibrous paper pulp material to adsorbent. For example, membranes having an upper layer containing a large quantity of adsorbent and a lower layer containing fibrous paper pulp material alone, membranes having upper and lower layers containing a large quantity of fibrous paper pulp material and having therebetween an intermediate layer comprising an adsorbent, or membranes further having an outer layer of thin paper and having therebetween an interlayer comprising fibrous paper pulp material and adsorbent serve to prevent the adsorbent contained in the membranes from escaping during transportation or use.

The oil regenerated filtration membrane of the present invention may be used in a sheet form as a filter medium to be placed on a porous bottom plate of a filter. In addition, this membrane may be made into a cup form to conduct filtration and purification through the whole wall of the cup. Further, several sheets of membranes may be bound to each other to form a cup. In this case, the membrane-binding materials are preferably oil-impermeable and flexible sheets, such as regenerated cellulose film, polyamide resin film, polycarbonate film, etc.

In the case of placing the sheet-like membrane at the bottom of a filter, it is extremely difficult to completely prevent leakage of an oil through around the membrane even when periphery of the membrane is shaped to be sufficiently fixed so that no openings exist, and part of the oil leaks through around the membrane without passing through it, resulting in insufficient adsorption and purification. When the membrane is formed or made into a cup, an oil added to the cup inevitably passes through the membrane constituting the wall of the cup, and thus the oil is completely regenerated. However, such cups are bulky and are disadvantageous because they require extra space for transportation, storage, and sale. This disadvantage can be overcome by folding cups made by binding several sheets using a flexible sheet which is less bulky as shown in the drawings.

The size of oil regenerating cup can be from 0.5 to 10 liters in volume; however, for household use, a size of from 1.0 to 1.2 liters is preferable in view of handling easiness. The thickness of cup must be made thicker as the volume of the cup increases, from the point of view of regeneration efficiency and strength, and is usually within the range of from 0.1 to 3.0 mm.

The shape of oil regenerating cup is not particularly limited, but the mouth of the cup is preferably large enough to facilitate pouring of a used oil from a frying pan. For example, inverted shapes of a circular cone, an elliptic cone, a pyramid, or corrugated form thereof, inverted shapes of truncated cone or pyramid having a bottom, a bottomed cylinder or a bottomed square pipe, or other arbitrary shapes may be employed.

The optimum temperature of the edible oil to be purified by using the membrane of the present invention somewhat varies depending upon the characteristics of adsorbent and the kind of oil. However, in general, a higher temperature provides better regeneration efficiency as long as the oil is not decomposed or chemically changed. In general, temperatures between 80°C and 160°C are optimal. Since the temperature employed for frying is usually about 180°C, a frying oil is preferably poured into the cup immediately after frying taking it into consideration that the oil undergoes reduction in temperature upon pouring it into a filter or a regeneration cup to the above-described temperature range. In the case of regenerating a deteriorated oil after it has cooled, it is preferable to maintain the oil temperature at a level of 60°C or higher, because such temperature serves to reduce the oil viscosity, accelerate the filtration rate, and raise adsorption and purification efficiency.

A filter using the oil regenerating cup of the present invention does not always require a retainer (for example, the cup may be supported by a ring-shaped supporter) but, in the case of using the filter, the filter may be retained over a regenerated oil receiver (preserve vessel), so that transfer of the regenerated oil to another vessel is not necessary, thus handling of the oil becomes easier.

And, it is possible to improve the adsorption efficiency and filtration rate by maintaining the temperature of the oil to be regenerated at at least some definite level, which can be realized by providing the retainer itself with a heating device or by placing the retainer in a Dewar vessel.

The present invention will now be described in more detail by the following non-limiting

examples.

EXAMPLE 1

10 g of cotton pulp, 30 g of activated clay (Galleon earth V2R, made by Mizusawa Industrial Chemicals Ltd.), and 1,200 g of deionied water were added to a mixer (1.5 liters in volume), and mixed for 5 minutes.

Then, a cup-forming porous metal mold (120 mm φ in upper diameter, 100 mm φ in bottom diameter, 110 mm in height, and 1 mm φ in pore diameter) was placed on a suction tank, and the above-described mixture was suction-adsorbed on the metal mold to dehydrate it by bringing the outer portion of the porous metal mold into a vacuum state using a suction pump. Thus, there was formed an inverted head-cut conical cup.

Further, the wet state cup (adsorbed on the porous metal mold) was dried at 110°C for 30 minutes in a drier to remove moisture, then cooled to room temperature followed by removing it from the metal mold to obtain an edible oil regenerating cup.

Additionally, the cup had a weight of about 30 g and an average thickness of 0.8 mm.

800 g of an oil used for frying (salad oil; used for making fritters of vegetable, fish, and meat each twice) was poured into the thus-formed oil regenerating cup at 160°C to conduct oil regeneration.

The light transmittance and odor of the starting oil, sample oil for regeneration (deteriorated oil), and the regenerated oil are shown in Table 1.

EXAMPLE 2

In the same manner as in Example 1 except for using 10 g of cotton pulp, 20 g of the same activated clay as used in Example 1, 10 g of diatomaceous earth (made by Kanto Chemical Co., Inc.), and 1,200 g of deionized water, there was formed an oil regenerating cup. Regeneration of the same deteriorated oil as used in Example 1 was conducted.

Results of the regeneration are shown in Table 1.

EXAMPLE 3

In the same manner as in Example 1 except for using 8 g of cotton pulp, 2 g of rayon pulp (made by Toyo Spinning Co., Ltd.), 28 g of the same activated clay as used in Example 1, 7 g of bentonite (aluminosilicate hydrate; made by Kanto Chemical Co., Ltd.), and 1,200 g of deionized water, there was formed an oil regenerating cup. Regeneration of the same deteriorated oil as used in Example 1 was conducted.

Results of the regeneration are shown in Table 1.

EXAMPLE 4

In the same manner as in Example 1 except for using 8 g of cotton pulp, 23 g of the same activated clay as used in Example 1, 8 g of aluminum oxide gel (made by Kyowa Chemical Industry Co., Ltd.), and 1,200 g of deionized water, there was formed an oil regenerating cup. Regeneration of the same deteriorated oil as used in Example 1 was conducted.

Results of the regeneration are shown in Table 1.

EXAMPLE 5

A high quality paper (made by Mitsubishi Paper Mills, Ltd.; 40 μ in thickness; 9.2 g/m^2 in basis weight) was adsorbed inside a cup-forming porous metal mold as in Example 1.

A mixture of 10 g of cotton pulp, 30 g of the same activated clay as used in Example 1, and 1,200 g of deionized water was suction-adsorbed inside the above-described woodfree paper. Other procedures were conducted in the same manner as in Example 1 to form an oil regenerating cup. Regeneration of the same deteriorated oil as used in Example 1 was conducted.

Results of the regeneration are shown in Table 1.

EXAMPLE 6

In the same manner as in Example 1, except using 10 g of cotton pulp and 1,200 g of deionized water, there was formed a cup.

Further, a mixture of 10 g of cotton pulp, 30 g of the same activated clay as used in Example 1 and 1,200 g of deionized water was suction-adsorbed inside the above-described cup to form a two-layered cup.

Additionally, the cup had a weight of 25 g and an average thickness of 1.5 mm.

Results of the oil regeneration are shown in Table 1.

COMPARATIVE EXAMPLE 1

60 g of the same activated clay as used in Example 1 was added to 800 g of the same oil (cooled to 160°C) as in Example 1 that had been used for making fritters, and mixed for 10 minutes using a stirrer. Then, the mixture was filtered through a filter paper (Toyo filter paper for Qualitative analysis No. 2, made by Toyo Roshi Co., Ltd.) set on a glass funnel to obtain a regenerated oil.

Results thus-obtained are shown in Table 1.

COMPARATIVE EXAMPLE 2

In the same manner as in Example 1 except for using 10 g of cotton pulp and 1,200 g of deionized water, there was formed a cup. Regeneration of the same deteriorated oil as in Example 1 was conducted.

Results thus-obtained are shown in Table 1.

TABLE 1

		Starting Oil	Deteriorated Oil	Regenerated Oil
Example 1	Light Transmittance (%)	87.9	37.0	76.2
	Odor	No	Yes	No
Example 2	Light Transmittance (%)	87.9	37.0	82.8
	Odor	No	Yes	No
Example 3	Light Transmittance (%)	87.9	37.0	84.5
	Odor	No	Yes	No
Example 4	Light Transmittance (%)	87.9	37.0	89.2
	Odor	No	Yes	No
Example 5	Light Transmittance (%)	87.9	37.0	79.0
	Odor	No	Yes	No
Example 6	Light Transmittance (%)	87.9	37.0	87.5
	Odor	No	Yes	No
Comparative Example 1	Light Transmittance (%)	87.9	37.0	93.3
	Odor	No	Yes	No
Comparative Example 2	Light Transmittance (%)	87.9	37.0	51.2
	Odor	No	Yes	Yes

Light transmittance in Table 1 is that for 450 mμ light measured by means of an automatic recording spectrophotometer (made by Hitachi, Ltd.) by placing a sample and a blank (distilled water) in a 10 x 10 x 30 mm quartz cell, and odor was sensed at room temperature, wherein when the sample oil possessed an odor different from that of the starting oil, the existence of odor of the sample oil is expressed as "yes."

As is clear from Table 1, characteristic values obtained in Examples 1 to 6 of the present invention were essentially the same as those in Comparative Example 1 and better than those in Comparative Example 2.

Additionally, Comparative Example 1 required complicated procedures because of collecting the activated clay by filtration through the filter paper.

As to other characteristics, acid value (AV), peroxide value (POV), and foaming properties were measured and, as a result, the cups of the present invention proved to give much better results than commercially available oil filters. The results are shown in Table 2 below.

TABLE 2

		Starting Oil	Deteriorated Oil	Regenerated Oil
Example 1	AV	0.04	4.56	0.88
	POV	1.66	78.50	2.50
	Foaming	10	60	14
Example 2	AV	0.04	4.56	1.00
	POV	1.66	78.50	3.65
	Foaming	10	60	15
Example 3	AV	0.04	4.56	0.95
	POV	1.66	78.50	3.80
	Foaming	10	60	16
Example 4	AV	0.04	4.56	0.65
	POV	1.66	78.50	2.00
	Foaming	10	60	13
Example 5	AV	0.04	4.56	1.00
	POV	1.66	78.50	5.00
	Foaming	10	60	16
Example 6	AV	0.04	4.56	0.35
	POV	1.66	78.50	2.30
	Foaming	10	60	15
Comparative Example 1	AV	0.04	4.56	0.68
	POV	1.66	78.50	2.30
	Foaming	10	60	14
Comparative Example 2	AV	0.04	4.56	4.50
	POV	1.66	78.50	75.00
	Foaming	10	60	54

Notes

AV (Acid Value, KOH mg/g): measured according to Shibsted method.
POV (Peroxide Value, meq./kg): measured according to Wheeler method.
Foaming (mm): calculated by placing 40 ml of a sample oil in a graduated test tube (250 mm height, 25 mm diameter), elevating the temperature of oil to 240°C, immersing potato piece (10 x 10 x 10 mm) in the oil with the immersion depth of 2 mm and measuring the height of foams.

Additionally, in the case of making fritters using an oil regenerated according to the present invention, the fritters had the same color and taste as those made by using a fresh (starting) oil even though the odor of activated clay was slightly present in some cases.

EXAMPLE 7

10 g of cotton pulp, 30 g of the same activated clay as used in Example 1, and 1,200 g of deionized water were placed in a mixer (1.5 l in volume), and mixed for 5 minutes.

Then, a sheet-forming porous metal mold (300 mm in length, 600 mm in width, and 1 mm φ in pore size) was provided on a suction tank, and the above-described mixture was suction-adsorbed on the metal mold to dehydrate and form a sheet by applying vacuum to the bottom of the porous metal mold using a suction pump.

Further, the wet state sheet (adsorbed on the porous metal mold) was left in a drier (at 110°C) for 30 minutes to remove moisture, then cooled to room temperature and removed from the metal mold.

The resulting sheet was cut into trapezoidal pieces 1 and 1' (200 mm in upper side, 100 mm in lower side, and 160 mm in height), and adhesively bound to each other at both the sides and bottoms thereof by a binding material 2 of regenerated cellulose tape (20 mm in width and 20 μ in thickness) using a 5% methyl cellulose aqueous solution to form a cup as illustrated in Figures 1, 2, and 3.

The cup had a weight of 40 g and an average thickness of 1.5 mm.

600 g of an oil that had been used for making fritters was cooled to 160°C and poured into the above-described oil regenerating cup to conduct regeneration.

Light transmittance and odor of the starting oil, sample oil for regeneration (deteriorated oil), and regenerated oil are shown in Table 3.

EXAMPLE 8

A sheet was formed in the same manner as in Example 7, and regeneration of a deteriorated oil was conducted in the same manner as in Example 7, except that the sheet pieces were welded by hot-pressing using as a binding material a 12-μ thick polyamide resin (nylon 6) film.

Results of the regeneration are shown in Table 3.

EXAMPLE 9

A sheet was formed in the same manner as in Example 7, and regeneration of a deteriorated oil was conducted in the same manner as in Example 7, except that the sheet pieces were welded by hot-pressing using as a binding material a 12-μ thick polyethylene terephthalate film.

Results of the regeneration are shown in Table 3.

EXAMPLE 10

A sheet was formed in the same manner as in Example 7, and regeneration of a deteriorated oil was conducted in the same manner as in Example 7, except that the sheet pieces were welded by hot-pressing using as a binding material a 12-μ thick polycarbonate film.

Results of the regeneration are shown in Table 3.

TABLE 3

		Starting Oil	Deteriorated Oil	Regenerated Oil
Example 7	Light Transmittance (%)	87.9	37.0	83.6
	Odor	No	Yes	No
Example 8	Light Transmittance (%)	87.9	37.0	81.6
	Odor	No	Yes	No
Example 9	Light Transmittance (%)	87.9	37.0	80.0
	Odor	No	Yes	No
Example 10	Light Transmittance (%)	87.9	37.0	78.8
	Odor	No	Yes	No

As to other characteristics, acid value (AV), peroxide value (POV) and foaming properties were measured and, as a result, the cups of the present invention proved to give much better results than commercially available oil filters. The results are shown in Table 4 below.

TABLE 4

		Starting Oil	Deteriorated Oil	Regenerated Oil
Example 7	AV	0.04	4.56	1.00
	POV	1.66	75.50	3.20
	Foaming	10	60	15
Example 8	AV	0.04	4.56	1.00
	POV	1.66	75.50	3.60
	Foaming	10	60	15
Example 9	AV	0.04	4.56	1.20
	POV	1.66	75.50	4.00
	Foaming	10	60	16
Example 10	AV	0.04	4.56	1.45
	POV	1.66	75.50	4.80
	Foaming	10	60	17

AV, POV and Foaming: The same as defined in Table 2 above.

EXAMPLE 11

In order to overcome transfer of clay odor observed in Examples 1 to 10, deodorizing of activated clay was conducted as follows.

500 g of the same activated clay as in Example 1 and 3 liters of deionized water were placed in a 10-liter beaker, boiled for 5 minutes and, after cooling, a 1/10 N sodium carbonate solution was added thereto in an amount of 0 ml, 100 ml, 400 ml or 600 ml.

The thus-treated suspensions were filtered using filter paper, and cakes obtained on the filter paper were dried at 120°C for 3 hours.

Cups (1.5 mm in thickness) as in Example 1 were formed using the thus-obtained activated clays, and regeneration of a deteriorated oil was conducted as in Example 1 using the cups. Then, vegetables (burdocks and carrots) were fried using the thus-regenerated oils to check for transfer of clay odor to vegetables. Light transmittance decreased little by little as the amount of added sodium carbonate increased, as shown in Table 5, but activated clay smell was substantially removed.

Additionally, fish-like odor of the oil itself was not detected at all with every oil.

TABLE 5

Amount of Added Na_2CO_3	pH	Light Transmittance: Deteriorated Oil	Light Transmittance: Regenerated Oil	Transfer of Odor to Fried Products
(ml)		(%)	(%)	
0	3.2	26.2	86.4	Yes
100	3.7	26.2	81.3	Slight
200	4.1	26.2	79.8	No
400	5.0	26.2	76.3	No
600	6.4	26.2	68.8	No

While the invention has been described above in detail with reference to edible oil regeneration it is to be appreciated that the invention is applicable to any oils which become contaminated with solids and contain other contaminants which require removal by adsorption.

CLAIMS

1. An oil regenerating membrane comprising 5 to 95 parts by weight fibrous paper pulp material and 95 to 5 parts by weight adsorbent per 100 parts by weight of membrane.

2. A membrane as claimed in claim 1, comprising a plurality of layers which have different composition ratios of said fibrous paper pulp material to said adsorbent.

3. A membrane as claimed in claim 1 or 2, which is of a cup-like form.

4. A membrane as in claim 1 or 2, comprising a plurality of sheet-like oil regenerating layers bound to each other by oil-impermeable, flexible sheets at the side edges and bottoms of said membranes to form a cup.

5. A membrane as claimed in claim 1, 2, or 3, wherein said adsorbent comprises at least one member selected from granular or powdery activated clay, acid clay, diatomaceous earth, zeolite, silica gel, calcium oxide and activated carbon.

6. A membrane as claimed in claim 5, wherein said adsorbent is a montmorillonite series adsorbent that has been treated with an aqueous alkaline inorganic material solution.

7. An oil regenerating membrane substantially as hereinbefore described in any one of Examples 1 to 11.

8. An oil regenerating membrane substantially as hereinbefore described with reference to the accompanying drawing.

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